Senior Thesis

An Examination of Iron-bearing Concretions from the Ohio Till

by
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the requirments for the degree of
Bachelor of Science in Geology and
Mineralogy at The Ohio State University,
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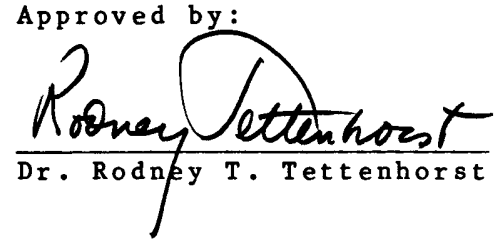
Approved by:



Dr. Rodney T. Tettenhorst

Acknowledgments

The author would like to thank Dr. Rodney T. Tettenhorst of The Ohio State University for suggesting and supervising this study. Thanks also to Jeff Swope and Ronald Sheets for their assistance and insights during the course of this study.

Table of Contents

INTRODUCTION

A concretion, collected by Mr. Michael Kukla near his home in Grandview, Ohio, a suburb of Columbus, which apparently came from a till deposit in central Ohio was obtained by Dr. Tettenhorst. The concretion had a very hard, brown outer shell with a soft yellowish interior, and was 9 cm by 3.5 cm in dimensions. X-ray analysis revealed the main constituents to be goethite, quartz, and lepidocrocite (FeOOH). This specimen was returned to its owner and was not made available to Dr. Tettenhorst for any further investigations. However, a few trips to the field proved successful in obtaining several concretions of very similiar appearance and mineralogical content, and these concretions were examined in this study. This paper is primarily concerned with the mineralogical analysis of these concretions and the reconstruction of a possible paleoenvironment based upon these analyses.

LITERATURE REVIEW

Concretions are defined as compact aggregates of mineral matter which are formed by the precipitation or segregation of mineral matter from a water solution around some type of nucleus in a sedimentary rock. Concretions generally are different in composition from the host rock in which they occur and represent a concentration of some minor constituent of that rock (Pettijohn, 1975).

Classifications

Several attempts have been made to classify concretions based upon different criteria. In 1903 Todd proposed a classification based on their mode of growth. He termed those concretions which grow from the center outward in a regular manner, accretions; those concretions which form by irregular and interstitial growth were termed intercretions; concretions which grow from the exterior inward were termed excretions; and concretions with a cylindrical form and a hollow core were termed incretions (Pettijohn, 1975). Todd's classification, however, was not widely accepted and is not in general use today.

Concretions and mineral segregations have also been classified on the basis of their form, internal structure, and composition. Classification by external form distinguishes between nodular and stratiform varities. Mineral aggregations classified on the basis of their internal structure may fall into one of four classes, 1) objects with irregular form and no regular internal structure, such as chert or flint nodules, 2) crystal aggregates with coarsely crystalline bodies such as rosettes and sand crystals, 3) mineral segregations such as fillings of cavities or fractures, for example, septarian nodules and geodes, and 4) a class of concretionary bodies which are normally subspherical, commonly very oblate to composite in form, and are precipitated from mineral solutions. These are simply termed concretions (Pettijohn, 1975).

Compositionally classified segregations include such objects as siliceous segregations (chert, flint), carbonates

(calcite, aragonite, siderite), phosphates, iron oxides, sulfates (gypsum, barite) and sulfides (pyrite, marcasite) (Pettijohn, 1975).

In 1971 Raiswell proposed a classification of concretions based upon their time of formation. In Raiswell's classification those concretions formed at the time of deposition of the enclosing sediment are termed syngenetic, those formed in the enclosing sediments while the sediments were still soft and unconsolidated are termed diagenetic, and those formed after consolidation of the enclosing sediment are termed epigenetic (Raiswell, 1971).

Origin and Emplacement

Determinimg the origin and manner of emplacement of concretionary bodies can present a problem since all concretions are not emplaced or developed in a similiar manner. Mineral segregations may make room for themselves by replacing the host rock, as do chert nodules, by deposition in pre-existing openings or fractures, as in geodes, by deposition in available pores around loci or centers in the host rock, as in many calcareous concretions, or they may make room for themselves by thrusting aside the enclosing matrix (Pettijohn, 1975).

Some criteria have been developed which may be applied to the problem of the time of emplacement of a concretionary body as addressed in Raiswell's classification. Chert and manganese nodules which are now forming on the seafloor are considered to be syngenetic and show no structure. Concretions which contain full bodied, uncrushed fossils suggest an early formation of the

concretion before consolidation of enclosing sediments and are therefore considered to be diagenetic. In 1927 Tomkieff provided criteria whereby the concretions could be dated relative to their host rocks using Raiswell's classification. Some concretions may show the passage of bedding planes, or laminae through the concretionary body. If at the margin of the concretion the laminae converge toward its major axis the concretion is assumed to have experienced compaction during the later stages of its growth, therefore, concretions containing deformed, converging laminae are assumed to have begun growth within unconsolidated sediments and to have been subjected later to compaction and deformation. These concretions are therefore classified as diagenetic (Raiswell, 1971).

Concretionary bodies which display the passage of laminae through the concretion without distortion of the concretion-matrix interface are considered to be epigenetic. Tomkieff (1927) demonstrated that the continuity of laminae through the concretionary body without distortion suggests a lack of compaction and therefore these concretions are considered to have formed after consolidation of the enclosing sediment, hence the term epigenetic (Raiswell, 1971).

During the growth of a concretion the two main controls of laminae configuration are 1) the length of growth span and 2) the rate of compaction (Raiswell, 1971). At shallow depths, with continuous deposition, a sediment compacts rapidly and a growing concretion will develop deformed laminae, whereas at greater depths compaction is slow and a concretion will develop parallel

laminae (Raiswell, 1971).

A post-depositional origin is suggested by concretions which display deformation of the enclosing rock as a result of the growth of the concretionary body, however, one must be careful to distinguish this from deformation caused by the compaction of the matrix about an already resistant body (Pettijohn, 1975).

Growth and Morphology

`Ironstone´ concretions frequently display a flattening along their major axis. This is attributed to their method of growth. Shortly after deposition conditions of permeability anisotropy may develop within fine-grained sediments and concretions may reflect this property of the sediment by preferential growth along the bedding planes (Raiswell, 1971). As a concretion grows the removal of ions from the pore waters for cement formation establishes concentration gradients around the concretion. Since the permeability across the sedimentary laminae is relatively high, diffusion is slow and growth normal to the bedding plane is retarded (Raiswell, 1971). The circularity of concretions along bedding planes reflects the uniform permeability along these bedding planes (Raiswell, 1971).

Mineralogy

Typical compositions for concretions frequently include either siderite or pyrite. Which one of these minerals forms may be a good indicator of the environment under which a concretion developed. The presence of siderite rather than pyrite in a

concretion suggests it developed in the presence of fresh water rather than brackish or marine water. Thermodynamic data on siderite growth (Curtis 1967) suggests that this mineral will only develop in an environment characterized by the absence of sulfide ions and a high partial pressure of carbon dioxide (higher than that in normal seawater saturated with calcium carbonate) Raiswell, 1971. Berner (1971) demonstrated theoretically that siderite is only stable relative to pyrite at extremely low dissolved sulfide activities (Postma, 1982). This suggests that if sufficient hydrogen sulfide is produced by bacterial sulfate reduction, the precipitation of pyrite and other iron sulfides will never allow ferrous iron concentrations to reach a level whereby siderite becomes stable (Postma, 1982). Since seawater contains large amounts of sulfate, reducing conditions inevitably result in hydrogen sulfate production and as a result siderite should not be stable in marine environments (Postma, 1982). Siderite is mainly found in fresh water sediments under these conditions; 1) low chloride levels for the pore waters, 2) no dissolved sulfides, 3) high ferrous iron concentrations, 4) relativly high partial pressures of carbon dioxide, and 5) Ca++/Fe++ much less than seawater (Postma, 1982).

However, siderite may not be considered a totally safe paleoenvironmetal freshwater indicator since siderite in minor amounts may possibly form in brackish or marine environments below the zone where sulfate reduction is complete if sufficient iron is available (Postma, 1982). The reduction of ferric oxyhydroxides by organic matter in the absence of sulfide

production results in the formation of siderite (Postma, 1982).

Lepidocrocite is not as common a mineral constituent of concretions as is siderite or pyrite, yet if present, lepidocrocite may also shed some light on the environmental conditions during the concretion's development.

Lepidocrocite normally forms from the oxidation of Fe(II) compounds produced by microbiological reduction and therefore occurs as a constituent of hydromorphic (water-saturated) soils in many regions (Schwertmann and Thalmann, 1976). However Tarzi and Protz (1978) found lepidocrocite associated with mica particles in two well drained Ontario soils developed on granite and granite-gneisses (Ross and Wang, 1979). They concluded that the Fe(II) required for lepidocrocite formation was probably derived from primary minerals such as biotite (Ross and Wang, 1979). Nonetheless lepidocrocite is most commonly associated with hydromorphic soils. Obviously lepidocrocite will not automatically form in anerobic soils with a Fe(II) content. Other factors which exert an influence on lepidocrocite formation are pH level, partial pressures of carbon dioxide, the rate of oxidation, presence of various dissolved compounds, and the existence of already nucleated species (Schwertmann and Thalmann, 1976).

Although lepidocrocite is a relativly stable mineral it may be further stabilized by organic compounds to the more stable polymorph goethite (Schwertmann, 1979). Therefore, the presence of goethite, if formed from lepidocrocite, may be misleading as an environmental indicator.

Sample Localities

The first concretion (#1) used in this study was collected by Mr. Michael Kukla. This concretion, pictured in Fig. 1, contains lepidocrocite as well as quartz and goethite and was originally assumed to have occurred in the Ohio Shale as many local concretions do. With the goal of obtaining additional concretions a trip was made to Reynoldsburg, Ohio to the property of Mr. and Mrs. Swope. A small creek runs through their property exposing the Ohio Shale along the banks and the creek bed. Two more concretions (#'s 2 and 3), shown in Fig. 2, were collected from this site which had a similiar appearance and mineralogical content, however, lepidocrocite did not occur in these concretions. The final collecting site was on the main campus of The Ohio State University where construction of a new chemistry laboratory was underway. Jeff Swope collected several concretions (#4), shown in Fig. 3, from the construction pit which had not yet reached bedrock. From this collecting site it became apparent that these concretions, and most probably the previously collected concretions, were floating in glacial till rather then occurring in the Ohio Shale.

Hand Speciman Description

Concretion #1 measured 9 cm by 3.5 cm. This concretion has a yellowish interior surrounded by a brown rind (Fig. 1). The yellowish interior is speckled with tiny dark spots not identifiable with a 10x handlens. The interior is compact yet may be scratched by a fingernail and is massive showing no internal structure, such as laminae. The contact between the

core and the rind is quite sharp and distinct, the rind being about 0.5 to 1.0 cm in thickness. The rind is very hard and dense, too hard to be scratched with a fingernail. The rind appears to display layering, the entire rind apparently composed of four smaller layers. Each layer is similiar to the next and in places the layers are indistinguishable.

Concretions #'s 2, 3 & 4 are very similiar in size and appearance to # 1. They all have soft interiors relative to their hard, thin, outer rinds. These concretions are pictured in Figs. 2 & 3.

X-ray Analysis Procedures

Samples were removed from each concretion using a small pocket knife and ground with a pestle and mortar to a flour-like powder. The powder was then mixed with acetone and evenly applied on a glass slide and allowed to dry. Samples were removed from the inner core and outer rind of each concretion. Concretions #'s 2 & 3 also had samples removed from the core-rind contact.

The mineral analysis was accomplished using Philips XRG-3100 X-ray diffraction equipment. Settings on the equipment included 35 kilovolts, 15 miliamps, 500 counts/second range, scintillation detector, scanning speed of $2^{\circ}\,2\theta$ per minute, and the X-ray chart speed was 60 inches/hour. A theta compensating slit along with a graphite monochromater was used.

A total of eight patterns were run in an effort to decipher any mineralogical changes which may occur from the central core to the outer rind.

X-ray Analysis Results

X-ray analysis of the core of concretion #1 showed the mineral constituents to be quartz and goethite (Fig. 4a & 4b). The quartz peaks were sharp relative to the goethite peaks. The sharpness of a peak indicates the degree of crystallinity, the sharper the peak the higher the degree of crystallinity. Analysis of the rind showed quartz and goethite again plus the presence of lepidocrocite and possible a trace of K-feldspar (Fig. 5a & 5b).

Analysis of the inner core of concretion #2 showed the presence of siderite and quartz. The siderite pattern predominates over the quartz pattern and shows strong, sharp peaks whereas the quartz produced only two fairly small peaks (Fig. 6). Analysis of the outer section of the core showed the presence of siderite, quartz and a possible trace of calcite. Again the siderite peaks were strong and sharp in comparison to the quartz, and the calcite peaks were very weak (Fig. 7). The X-ray pattern for the rind showed the presence of siderite, goethite, quartz and a possible trace of hematite. The peaks on this pattern were in general broader and not as strong, perhaps indicating a lower degree of crystallinity (Fig. 8a & 8b).

The core of concretion #3 contained goethite, quartz and siderite, the quartz peaks being fairly strong compared to the goethite and siderite peaks (Fig. 9). The soft layer between the

core and the rind contained mainly quartz and siderite with some goethite and minor amounts of feldspar and mica/illite. The quartz and siderite peaks were much stronger and sharper then the goethite peaks (Fig. 10a & 10b). The outer rind displayed the prominence of quartz and siderite but also showed larger peaks of mica/illite, some kaolinite/chlorite, and traces of feldspar and possibly some hematite (Fig. 11a & 11b).

The patterns for concretion #4 showed the broadest peaks, the core constituents being mainly siderite with a trace of goethite, and the rind displaying goethite, siderite and quartz (Fig. 12a & 12b). Table I is a summary of the X-ray analysis results showing which minerals were detected and where.

Discussion of Results

One of the interesting aspects of these concretions is their appearance. They all have relatively soft interiors with a harder, denser rind. At first sight one might attribute these rinds to the effects of chemical weathering, however, if this were the case one might expect the rind to be softer than the interior, unless the weathering had lead to a significant enrichment in iron content without disaggregating the weathered portions. Another possibility for the presence of these rinds may be due to a change of conditions during the precipitation of the concretions. The results from concretion #1 seem to support this idea. The core of concretion #1 contains quartz and goethite while the rind contains mainly quartz, goethite and lepidocrocite. Lepidocrocite is not a weathering product of goethite but rather will precipitate instead of goethite under

the proper conditions. Goethite is the most frequently occurring form of iron oxide in soils, and it has been shown that thermodynamically it has the greatest stability under most soil conditions (Schwertmann and Taylor, 1977). Lepidocrocite forms from the oxidation of precipitated ferrous iron hydroxy compounds and appears to be restricted mainly to hydromorphic soils where the presence of ferrous iron is generated due to an oxygen deficiency (Schwertmann and Taylor, 1977). If not oxidized the ferrous iron may be precipitated as a ferrous compound or, more likely, as a mixed ferrous/ferric iron compound (Schwertmann and Taylor, 1977). The ferrous/ferric iron hydroxy compound is formed by a reaction of ferrous iron and ferrihydrite. At zero or low partial pressures of carbon dioxide its formation is favored by high pH, high total iron concentrations in solution, low oxidation rates, and, within certain limits, by high ferric/ferrous iron ratios (Schwertmann and Taylor, 1977). The ferric iron oxides are formed from these ferrous/ferric iron compounds by a topotactic (one or two phase) oxidative transformation sometimes including dehydration (Schwertmann and Taylor, 1977). The order and rate of these processes are the important factors in determining the type of ferric oxide formed, leading either to lepidocrocite or maghemite (Schwertmann and Taylor, 1977). With fast oxidation, the ferrous/ferric iron hydroxy compound will be fully oxidized before partial dehydration and lepidocrocite will be formed, however, the presence of high concentrations of carbon dioxide in the air during oxidation of a ferrous chloride solution leads to the

formation of goethite rather then lepidocrodite (Schwertmann and Taylor, 1977). Therefore it seems likely that during the initial precipitation of concretion #1 goethite was the stable iron mineral and near the end conditions changed allowing precipitation of both goethite and lepidocrocite. The most important factor which allows lepidocrocite to precipitate with goethite would be a lowering of the carbon dioxide content in the system, and therefore it may be assumed that weathering has apparently not played a significant role in the mineralogical changes observed in concretion #1.

None of the next three concretions examined contained lepidocrocite, rather they contained siderite and goethite as the main iron-bearing minerals. The absence of lepidocrocite in a calcareous environment agrees well with laboratory results which indicate that the introduction of carbon dioxide into the system favors the formation of goethite rather then lepidocrocite as previously discussed (Schwertmann and Taylor, 1977). However, these concretions have a very close resemblance to concretion #1 in that they also have fairly soft interiors with a hard outer rind, and again the question as to how this rind has formed, via chemical weathering or a change in conditons during precipitation, arises.

Concretion #2 was shown to contain siderite and quartz in its core and its rind was shown to contain quartz, goethite, siderite and some hematite. In a reducing environment either siderite, pyrite or magnetite will be the stable iron mineral depending on concentrations of sulfur and carbonate in the solution. Fig. 13 shows an Eh-pH diagram displaying the

stability fields of common iron minerals. A notable ommission from this diagram is a field for goethite which is certainly an important iron mineral in soils; its relation to hematite is shown by the reaction:

$$2FeOOH = Fe_2O_3 + H_2O \quad \text{(Krauskopf, 1979).}$$

The relative stability of the two minerals is evidently dependent on the vapor pressure of water which should, therefore, be another variable on the diagram, yet if this variable is represented the results will not be realistic because the hematite-goethite reaction is very slow and attainment of equilibrium is not assured, therefore, it seems best to ignore this reaction and simply note that the field for goethite on the Eh-pH diagram is approximately the same as that of hematite (Krauskopf, 1979). An increase in either Eh or pH or both would, therefore, favor the precipitation of goethite and or hematite over siderite. Based on this the mineralogical changes observed in concretion #2, a sideritic core with a rind containing goethite, siderite and hematite, may also be explained by a change of conditions during precipitation. This time an increase in either Eh or pH or both would result in the mineralogical changes observed and again weathering probably did not play a significant role in the concretion's mineralogy.

The third concretion, #3, contains both goethite and siderite in the core along with some minor feldspar, the rind, however, contains mainly siderite as the iron-bearing mineral. The disappearance of goethite may represent a lowering of Eh and pH values during precipitation favoring the formation of siderite

over goethite. A lowering of these values may also account for the apparent precipitation of K-feldspar in the core and the precipitation of kaolinite/chlorite and mica/illite in the rind. The following equation gives the relationship of K-feldspar to kaolinite when K-feldspar reacts with an aqueous solution:

$$2KAlSi_3O_8 + 9H_2O + 2H^+ =$$

$$Al_2Si_5O_5(OH)_4 + 4H_4SiO_4 + 2K^+ \text{ (Krauskopf, 1979).}$$

A lowering of the pH value will increase the H+ ion activity which will, by the Le Chatelier Principle, drive this equation to the right, thereby favoring kaolinite over K-feldspar.

Concretions #2 and #3 were collected from the same locality yet display two apparent different mineral successions. Concretion #2 showing mainly siderite in the core and goethite in the rind, and concretion #3 showing mainly goethite in the core and siderite in the rind. This may be most easily explained by assuming one concretion began precipitation under conditions favoring siderite and as conditions changed to favoring goethite the second concretion began precipitating. This fluctuation of conditions may have occured innumerable times, so similiar concretions may contain the same mineral constituents but the concentration of any one mineral in any one location of the concretion may only be determined experimentally.

The fourth concretion, #4, gave relatively poor X-ray patterns, however, it seems to show the same precipitation course as that of concretion #3, that is a sideritic core with a rind that has a high goethite content.

Conclusions

The concretions collected for this study apparently do not occur in the Ohio Shale as was originally assumed, therefore, the author can not draw any conclusions as to the conditions during the deposition of the shale as was originally intended. The concretions do, however, display mineralogical changes in cross section which allows one to draw conclusions on conditions during their precipitation. Based on the collecting localities it is assumed that these concretions are floating in the Ohio glacial till and, therefore, it may be possible to perform a future study to determine the source of these concretions based on the known movements of glaciers across Ohio.

The formation of these concretions seems to have included a change of conditions during precipitation which is reflected in the change of stable iron-mineral phases from the core to the rind.

Concretions #'s 2, 3 & 4 show the same basic changes with either siderite or goethite being the stable iron mineral phase in the core or rind. Concretion #1, however, displays lepidocrocite and a lack of siderite as would be expected since these two minerals do not occur in the same environment. This suggests that this concretion developed in an environment lacking carbon dioxide, whereas #'s 2, 3 & 4 developed in an environment which contained carbon dioxide.

Based on the mineral constituents of these concretions a few basic assumptions may be inferred as to conditions during

their precipitation.

In reducing environments the stable iron minerals may be pyrite, siderite, or magnetite, depending on concentrations of sulfur and carbonate in solution (Fig. 13), Krauskopf, 1979. Siderite occurrence is mainly restricted to neutral and basic solutions; it can precipitate from weakly acid solutions only if the concentration of dissolved iron is abnormally high (Krauskopf, 1979). Iron sulfides precipitate only under very reducing conditions, or from solutions in which the concentration of sulfide is unusually high. Therefore, iron carbonate precipitates in reducing freshwater environments, but probably not in present-day marine environments because seawater contains too much dissolved sulfur (Krauskopf, 1979). Siderite-bearing rocks frequently are interpreted in two ways, either as swamp deposits from freshwater basins, or as marine deposits formed at moderate depths in an ocean with less dissolved sulfur and more dissolved carbon dioxide than the oceans of modern times (Krauskopf, 1979).

The generally high concentration of siderite in these concretions suggests they most likely precipitated in a basic, reducing freshwater environment, possibly at the water-sediment interface where Eh and pH conditions could fluctuate periodically allowing different iron minerals to be the more stable phase at different times. This is reflected in these concretions by a change of mineralogy from the core to the rind, and the chemical weathering process is not believed to be responsible for the appearence or mineralogical variations observed.

Figure 1. Concretion #1

Concretion #2

Concretion #3

Figure 2

Figure 3. Concretion #4

CONCRETION	CORE	OUTER CORE	RIND
#1	quartz goethite	-	quartz goethite lepidocrocite feldspar (trace)
#2	siderite quartz	siderite quartz calcite (trace)	quartz goethite siderite hematite
#3	quartz goethite siderite	quartz siderite goethite feldspar mica/illite (trace)	quartz siderite mica/illite kaolinite/chlorite feldspar hematite
#4	siderite goethite	-	siderite goethite quartz

Table I

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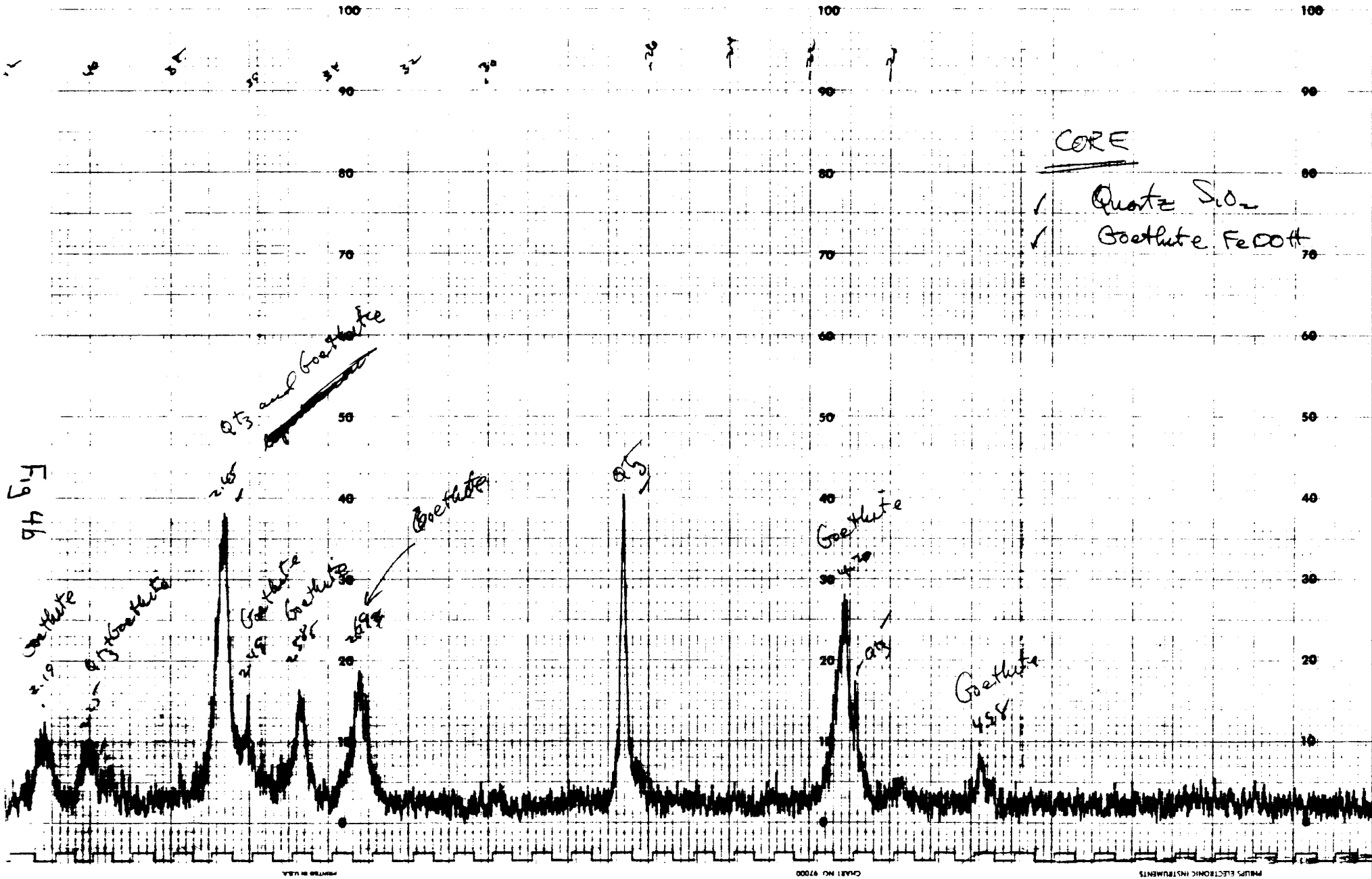
Fig 4a


CORE
Quartz SiO_2
Goethite FeOOH
Qtz and Goethite
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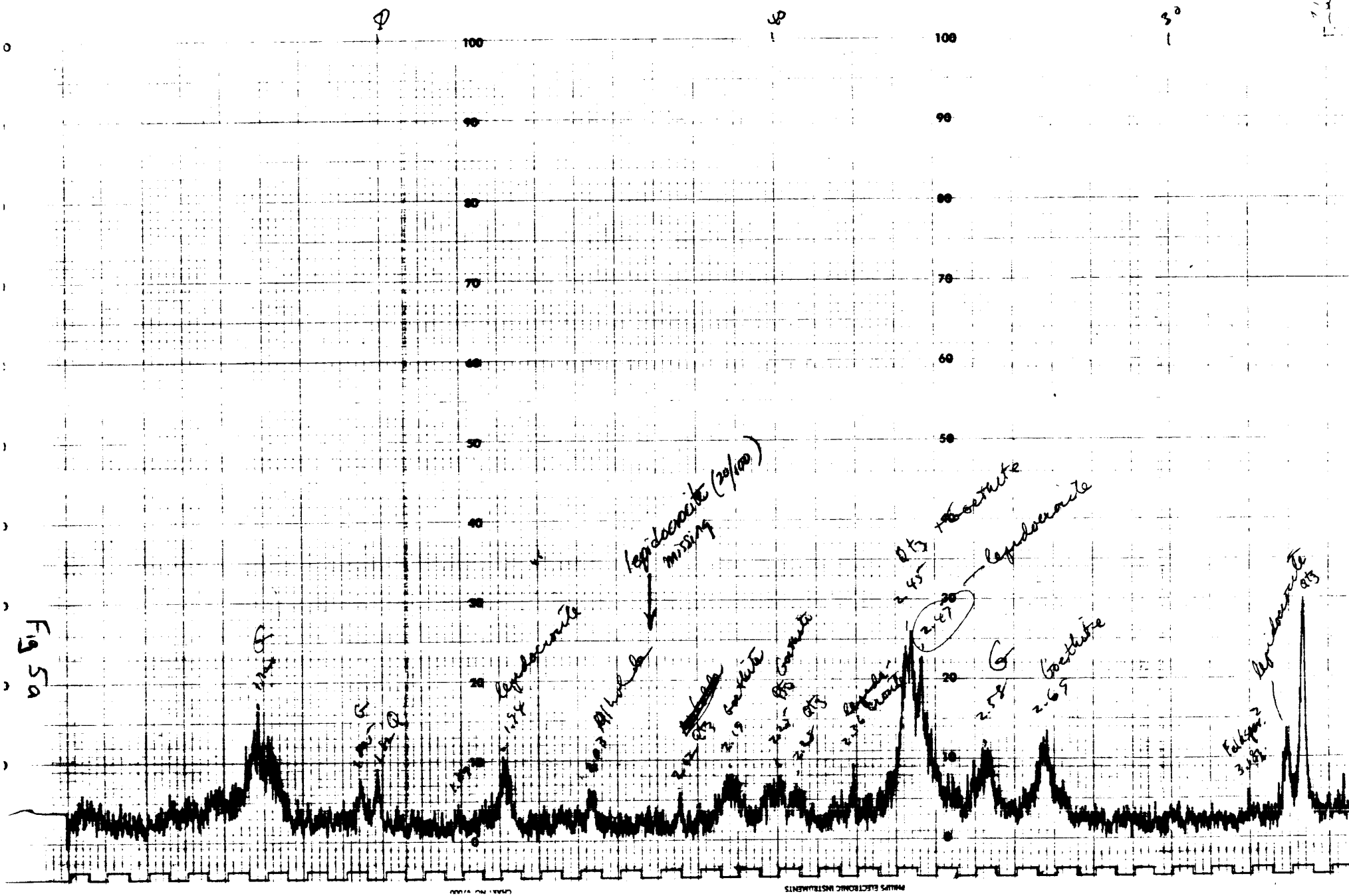
Fig 55
lepidocrocite (20/100)
missing
lepidocrocite
goethite
goethite
lepidocrocite
goethite
lepidocrocite
goethite
lepidocrocite
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Fig 56

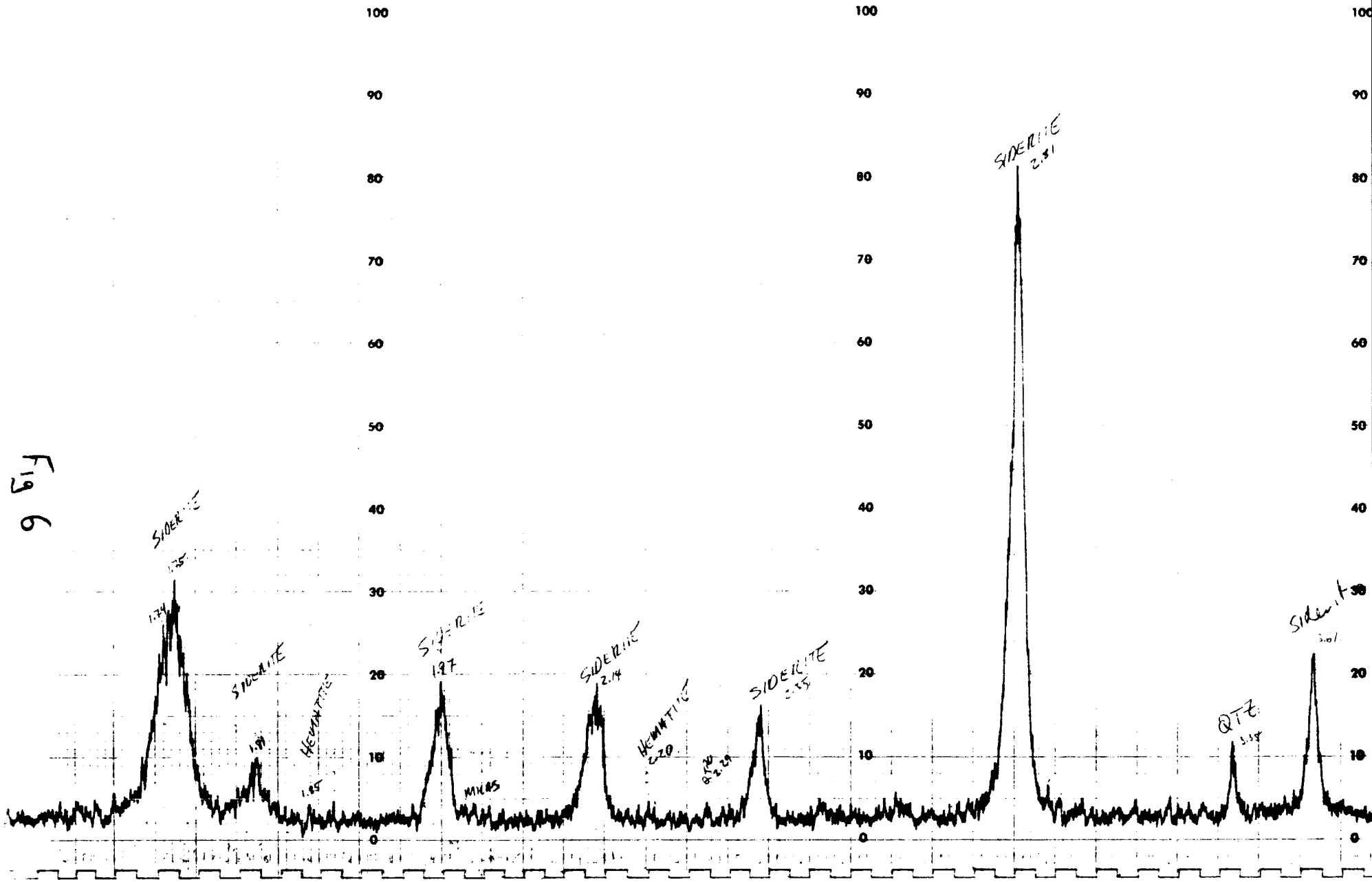
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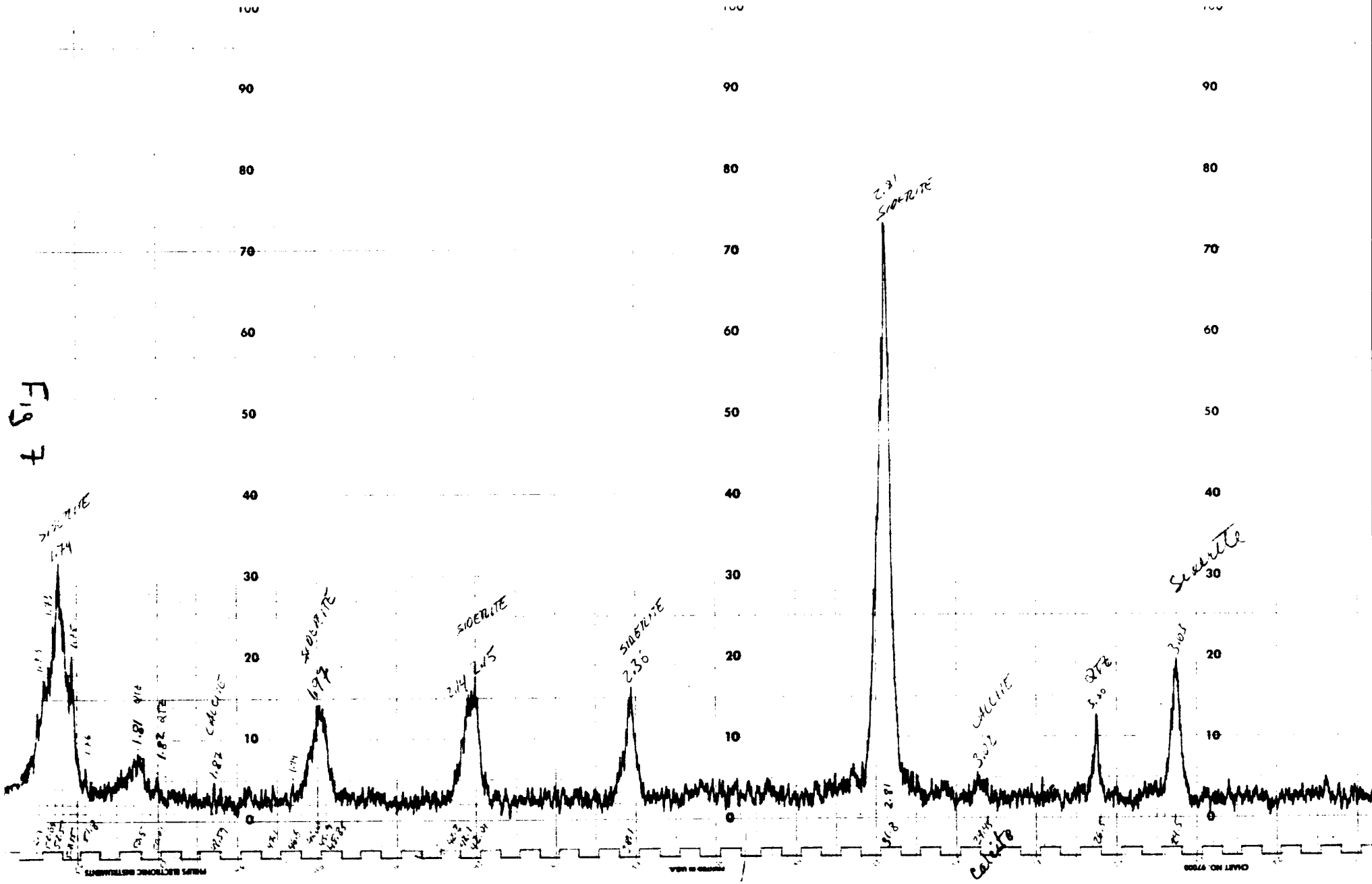
Fig 6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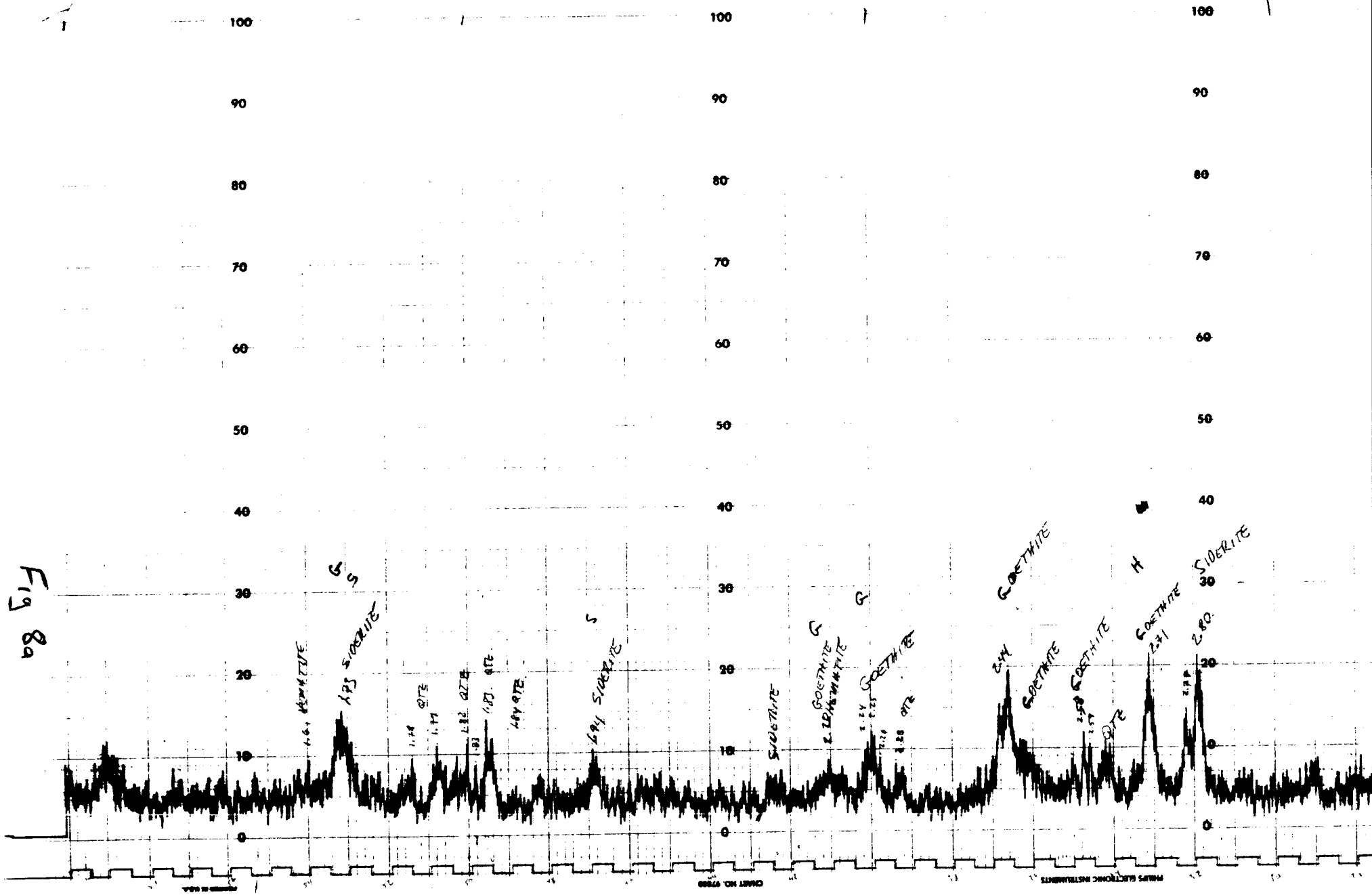
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Fig 8a

#1

OUTER RIND

Mainly: Quartz, Goethite
Some: Siderite
Maybe: Hematite

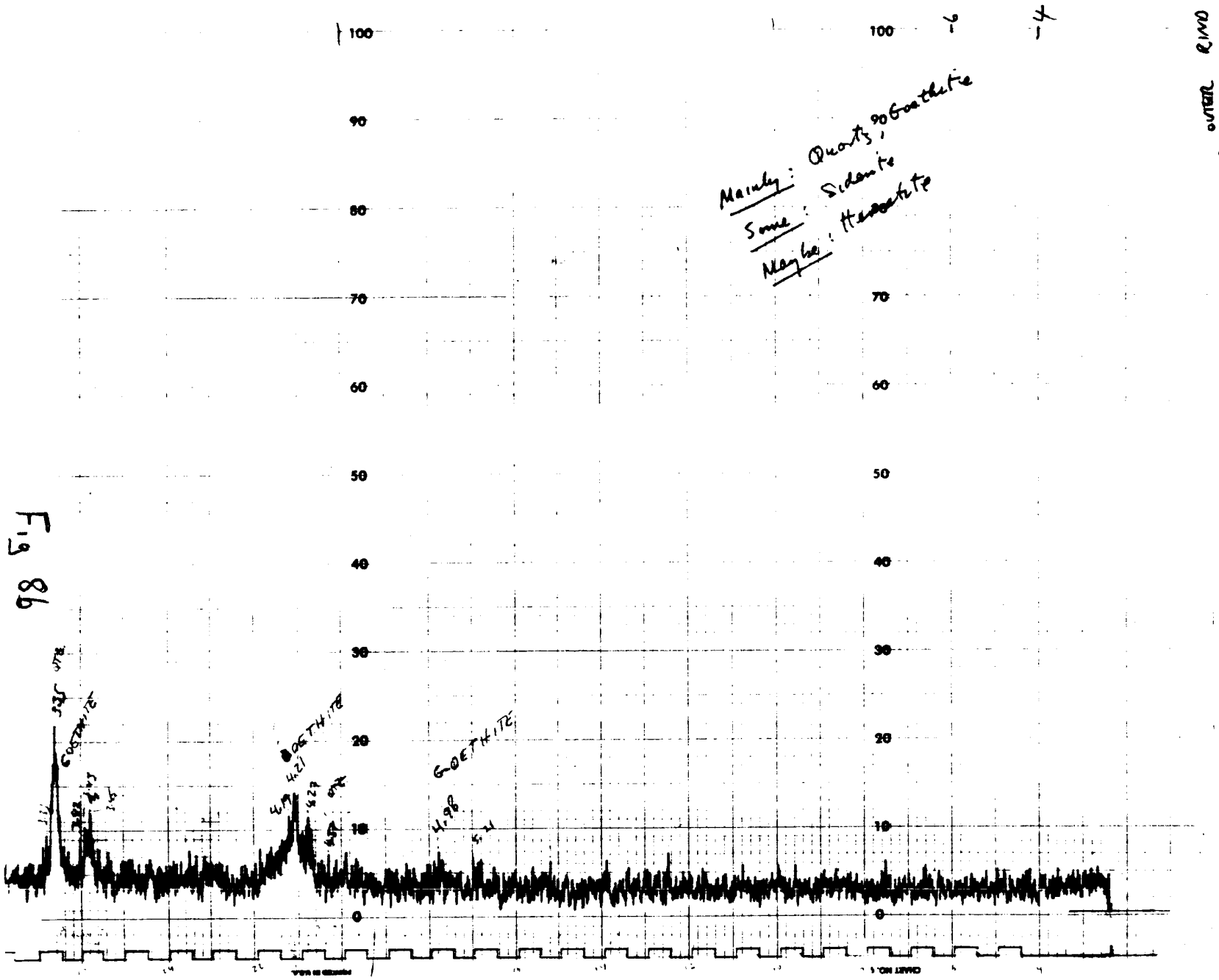

Fig 86

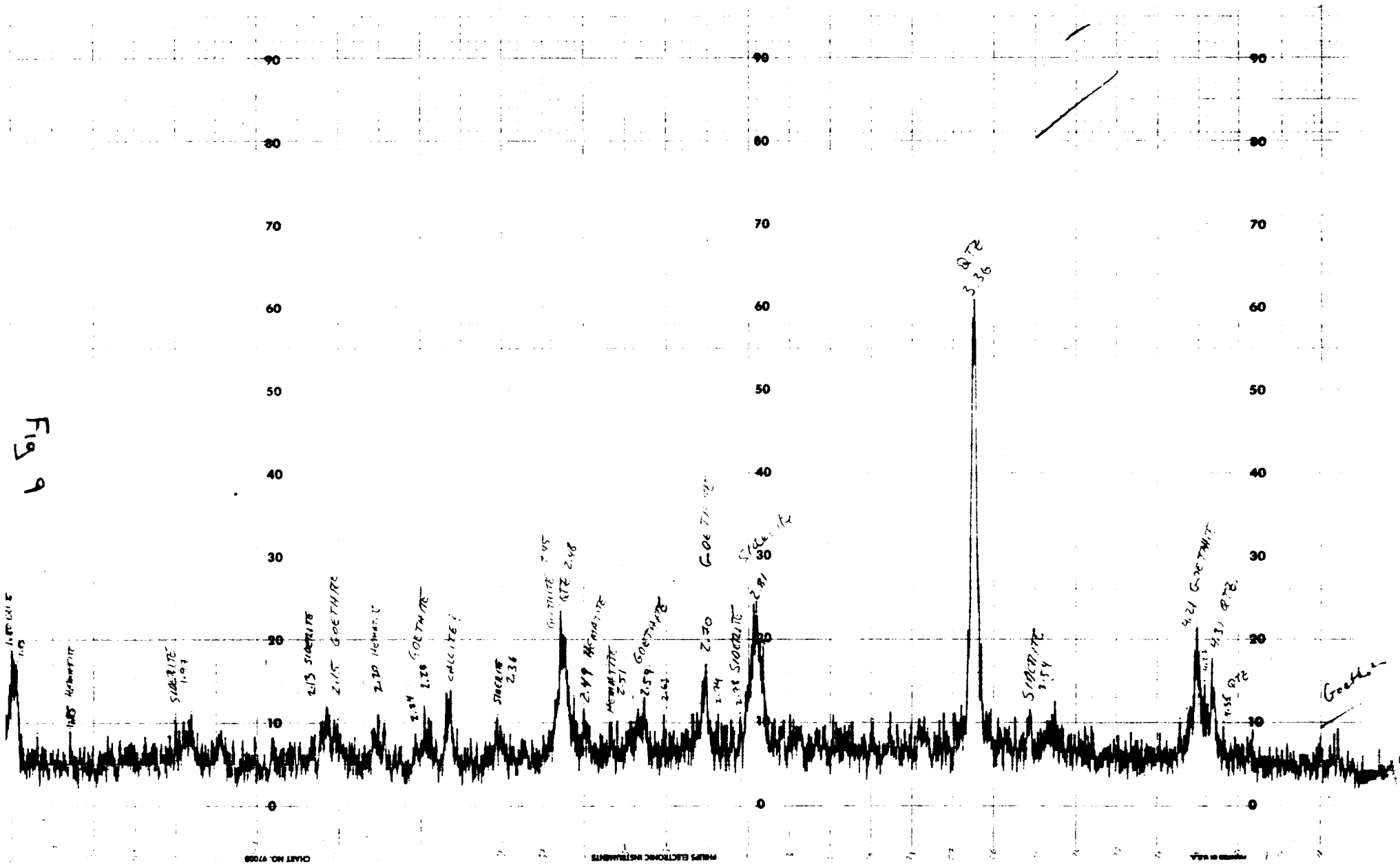
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Fig 9

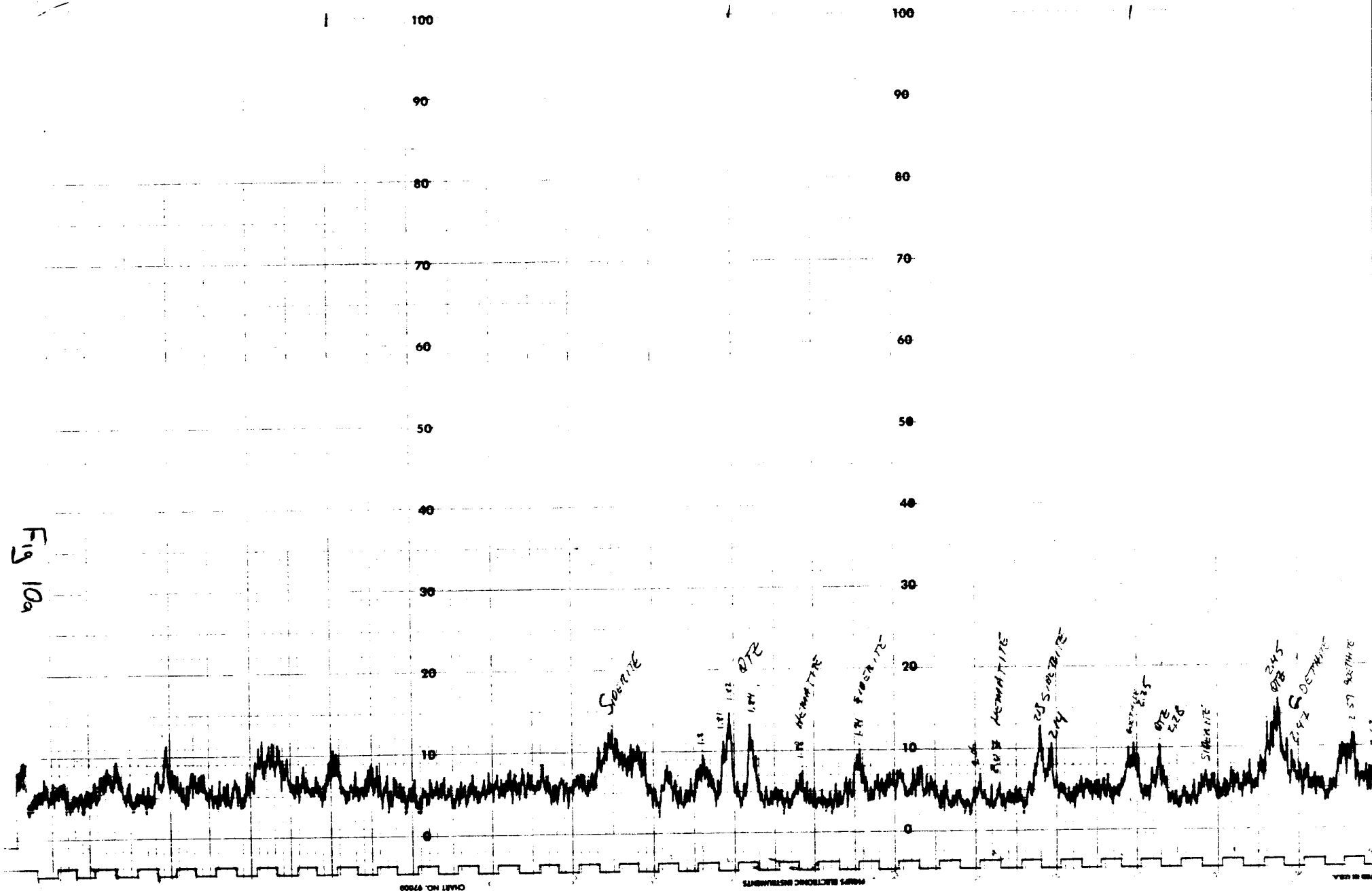
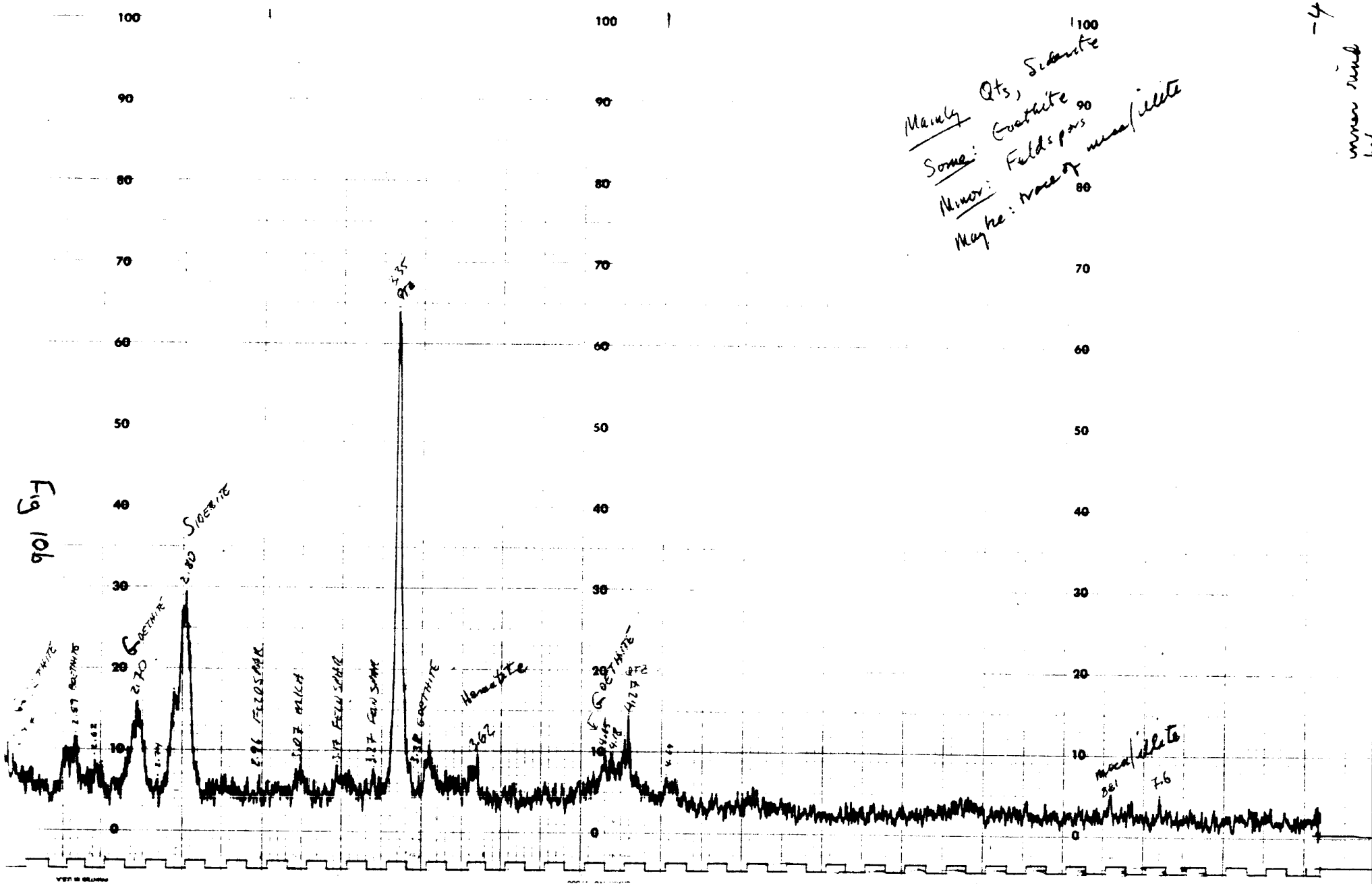

100
90
80
70
60
50
40
30
20
10
0
SIDERITE
QTZ
HEMATITE
HEMATITE
SIDERITE
QTZ
SIDERITE
QTZ
SIDERITE
QTZ
SIDERITE

Fig 10a


Mainly Qtz, Siderite
Some: Goethite
Minor: Feldspars
Maybe: trace of mica/illite
Fig 101
Siderite
Goethite
Hematite
Goethite
mica/illite
FELDSPAR
MICA
FELDSPAR
FELDSPAR
GOETHITE
GOETHITE
4.27
3.35
2.80
2.70
2.49
2.28
1.97
1.82
1.54
1.37
1.28
1.22
1.07
0.96
3.62
4.18
3.61
7.6

100
90
80
70
60
50
40
30
20
10
0
1.36 QTZ
1.82 RTZ
QTZ
HEMATITE
1.43 SIDERITE
SIDERITE
2.13 SIDERITE
2.28 QTZ
SIDERITE
2.46 QTZ
2.50 SIDERITE
2.49 HEMATITE

Fig 11a

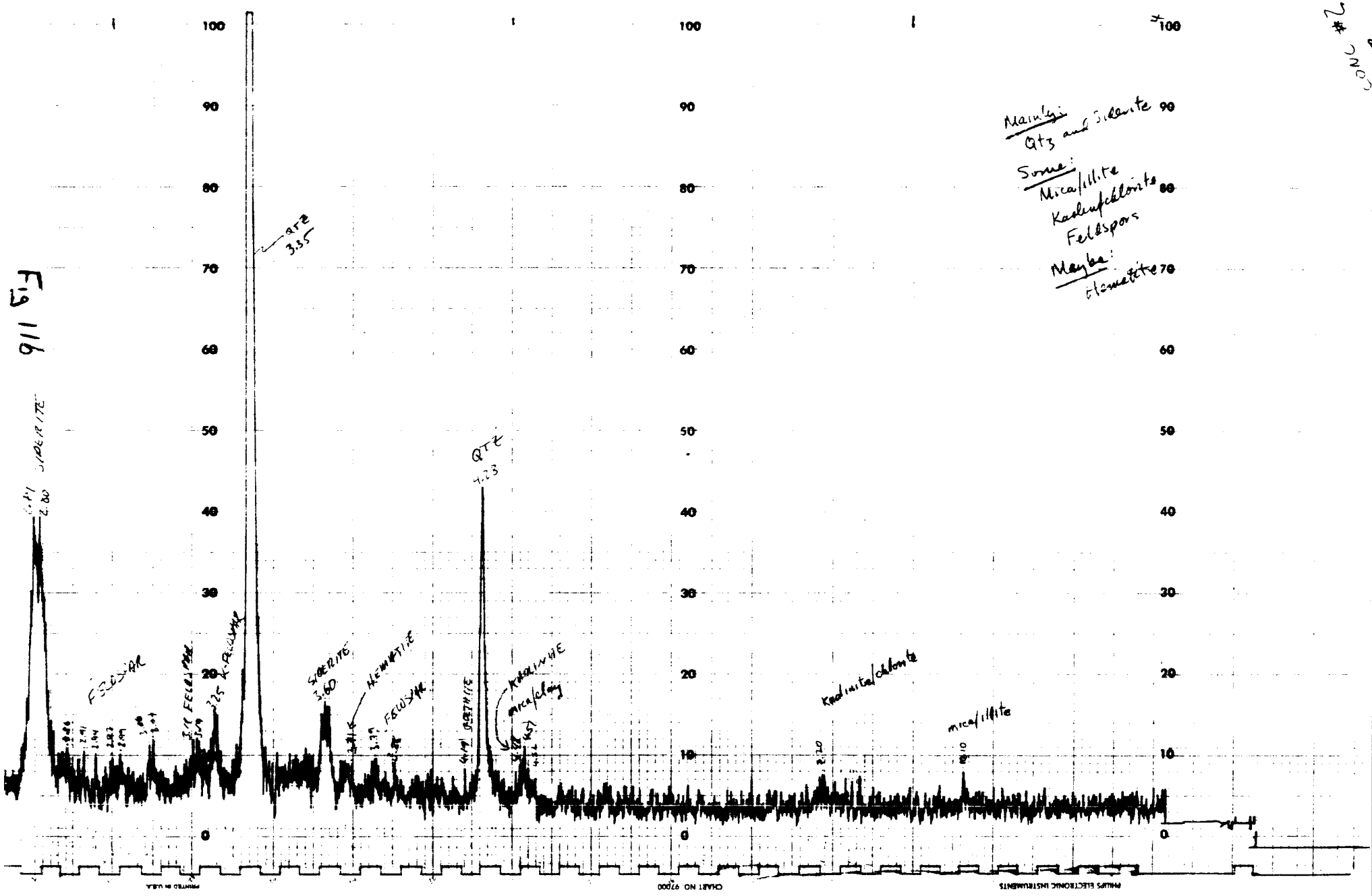


Fig 11b
Mainly
Qtz and Siderite
Some:
Mica/illite
Kaolinite/chlorite
Feldspars
Maybe:
Hematite
qtz
3.35
QTZ
4.23
SIDERITE
3.60
HEMATITE
FELDSPAR
KAOLINITE
mica/clay
Kaolinite/chlorite
mica/illite
100
90
80
70
60
50
40
30
20
10
0

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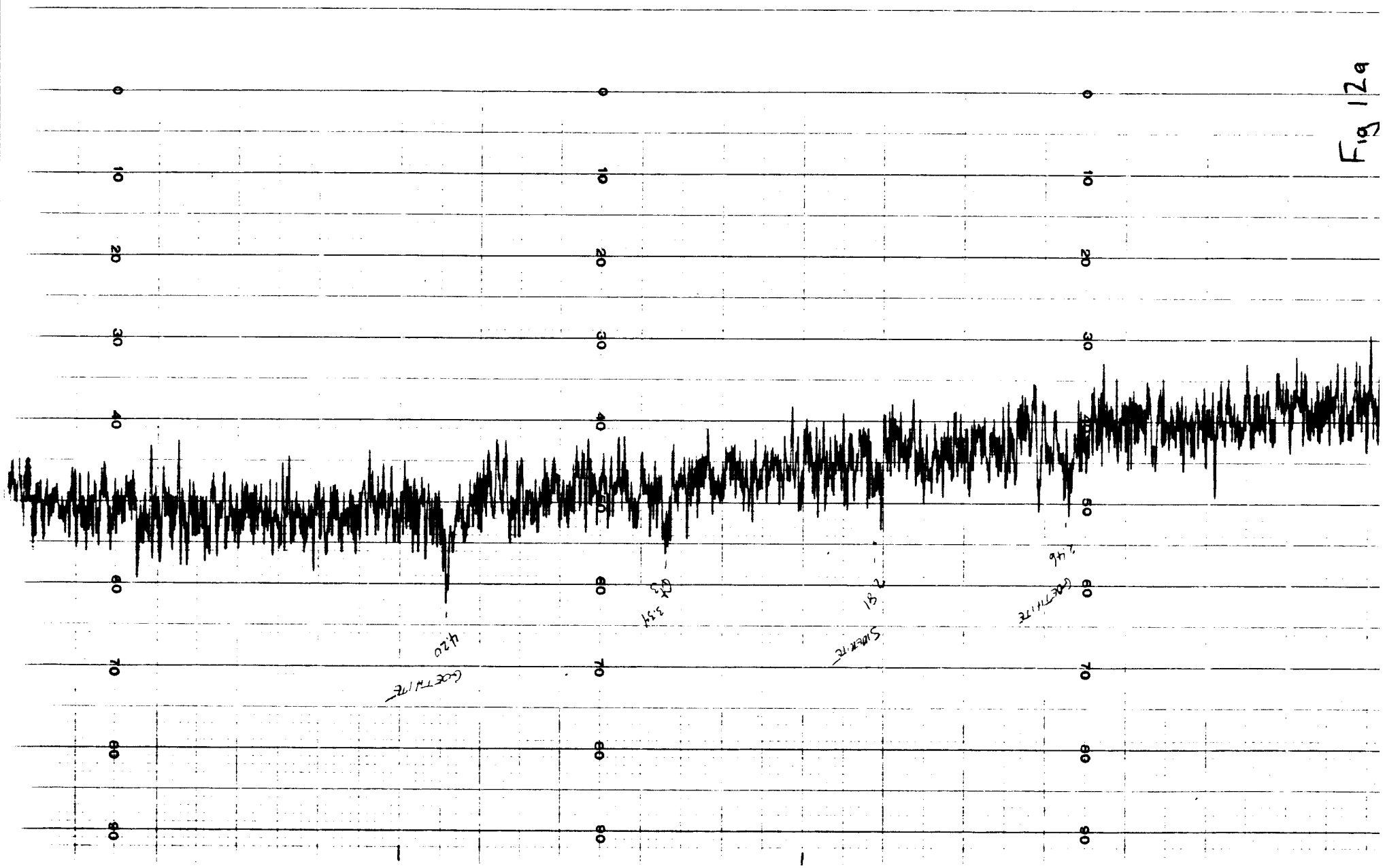
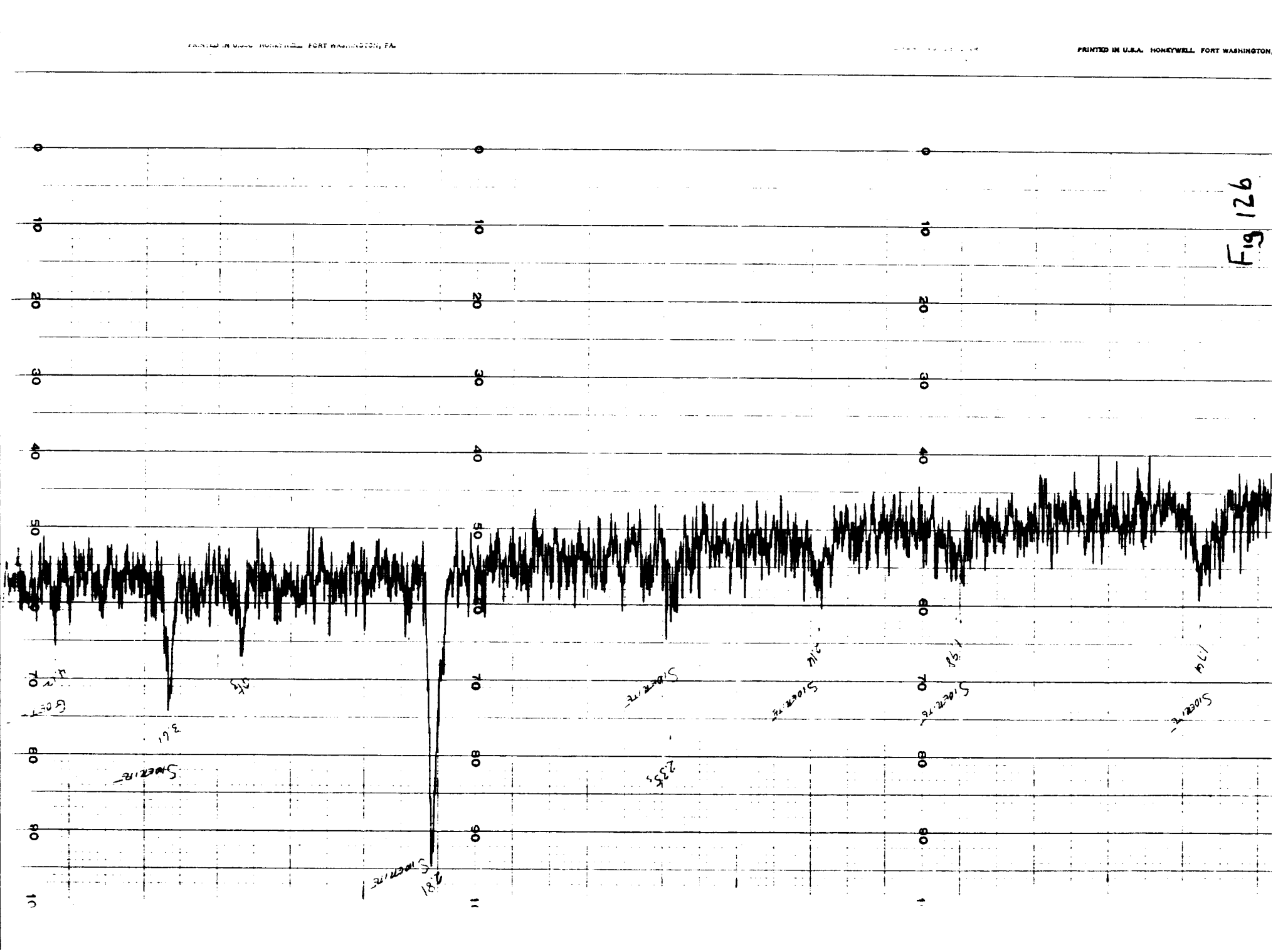


Fig 12a



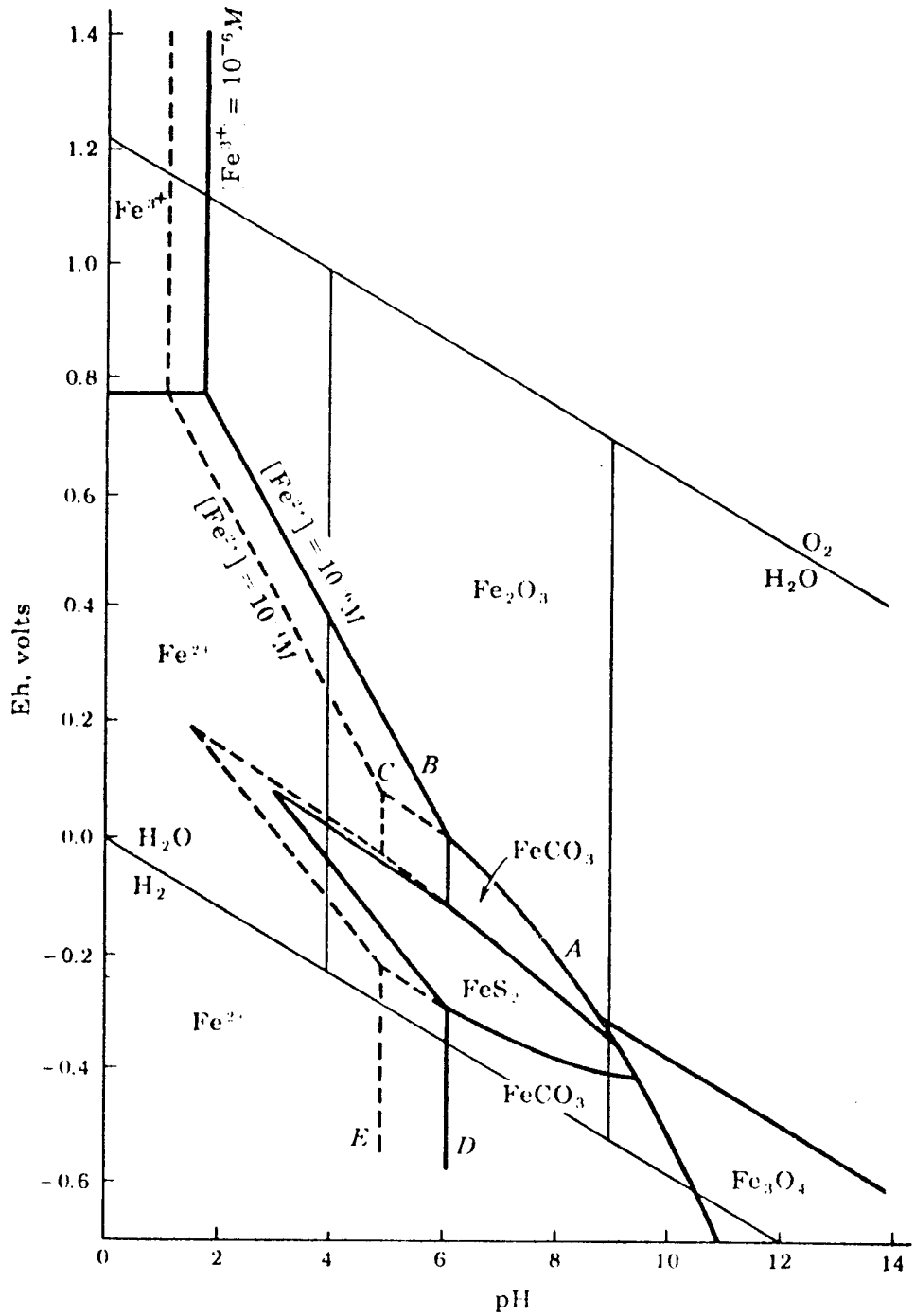


Figure 13 Eh-pH diagram showing stability fields of common iron minerals. Total activity of dissolved carbonate, $1M$, of dissolved sulfur, $10^{-6}M$. Solid field boundaries on left side of diagram are for total dissolved iron = $10^{-6}M$, dashed lines for $10^{-4}M$.

KRAUSKOPF, 1979

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